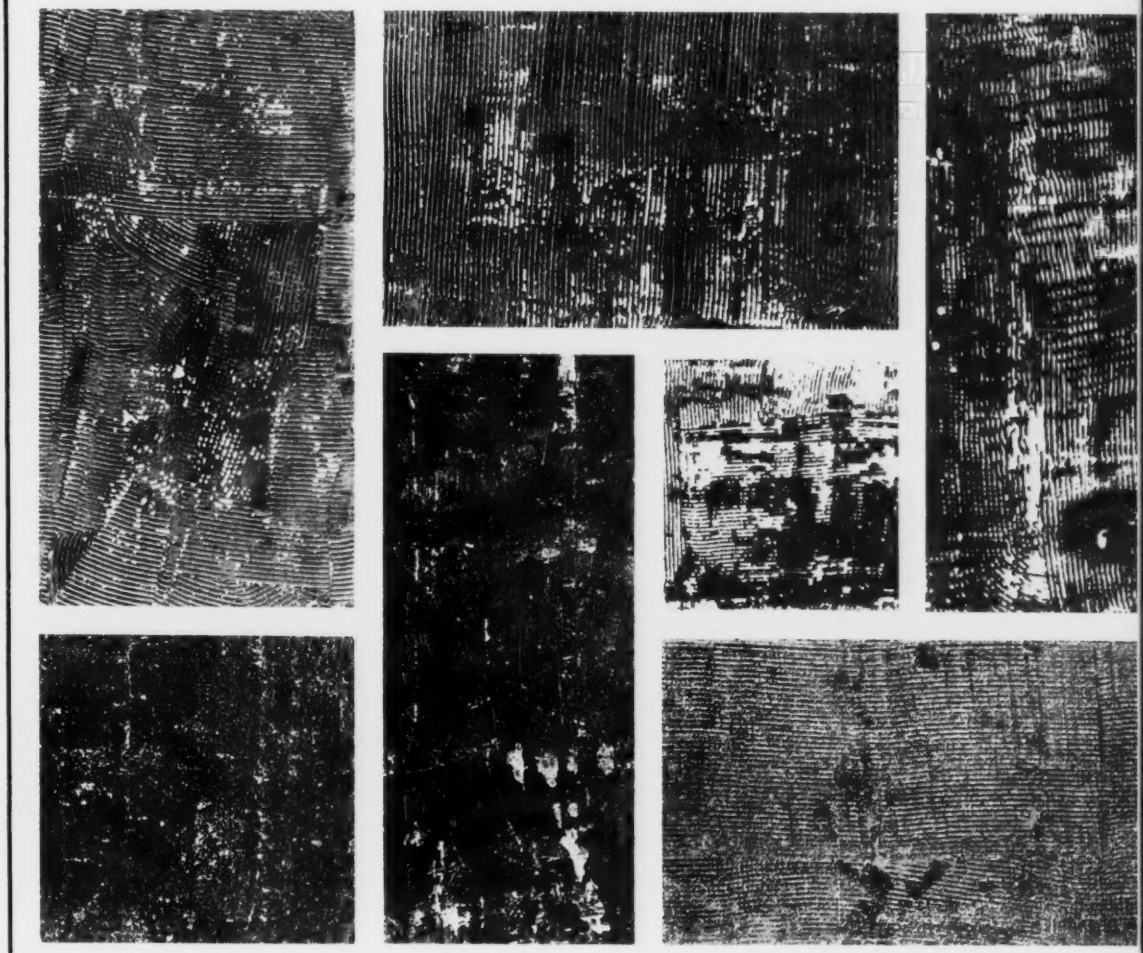
NATIONAL BUREAU OF STANDARDS April 1966

Technical News Bulletin



U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

Technical News Bulletin

APRIL 1966/VOL. 50, NO. 4/ISSUED MONTHLY

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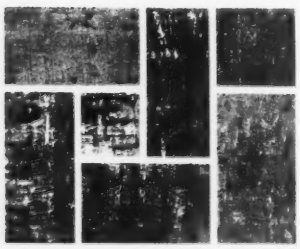

COVER

Design formed from photographs of experimental specimens that were used in a study of the bonding of tile to treated concrete (p. 58). The photographs showed concrete-specimen surfaces after removal of the tile.

U.S. DEPARTMENT OF COMMERCE
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The National Bureau of Standards serves as a focal point in the Federal Government for assuring maximum application of the physical and engineering sciences to the advancement of technology in industry and commerce. For this purpose, the Bureau is organized into three institutes—
- The Institute for Basic Standards
- The Institute for Materials Research
- The Institute for Applied Technology

The TECHNICAL NEWS BULLETIN is published to keep science and industry informed regarding the technical programs, accomplishments, and activities of all three institutes.

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This computer system accepts pictorial data and presents them in a manner meaningful to man. From left to right are shown: the FOSDIC microfilm scanner, its control, the data processor control, keyboard and punched tape inputs (in front of operator), and, in the background, logic and memory components.

Direct Communication Between MAN & COMPUTER

ACCESS System Will Process Emergency Data

ACCESS, a data-processing system developed by the NBS Institute for Applied Technology, represents an advance in communication between man and computers. This system will receive and process data from local and remote sources and present its output in a form immediately intelligible to the human operator.

ACCESS (so-called for *A*utomatic *C*omputer *C*ontrolled *E*lectronic *S*canning *S*ystem) was developed for use by the Office of Emergency Planning to help provide rapid access to digital and pictorial data. The ready availability of these data will aid the OEP in evaluating situations during a national emergency.

ACCESS accepts input information directly from microfilm records of hand-marked documents and digital information either from other machines or directly from its keyboard. It has been used in experimental work at the Bureau to accept such graphical material as specially prepared maps and charts. It will store the information, perform a variety of operations on it, and present outputs either in digital form for use by other machines or in a form requiring no further translation for man. The system includes an X–Y plotter which prepares such output displays as maps, charts, and diagrams.

The ACCESS System

ACCESS is primarily a data-gathering and data-preparation system, accepting marked documents which it scans by means of an advanced version of FOSDIC. The subsystem, *F*ilm *O*ptical *S*ensing *D*evice for *I*nput to *C*omputers, was initially developed jointly by NBS and the Bureau of the Census for machine reading of census documents.[1] The supervisory function of ACCESS was derived from the AMOS IV (*A*utomatic *M*eteorological *O*bservation *S*tation) system [2] developed by NBS and the Weather Bureau for storing and processing weather data from remote stations.

ACCESS is intended to control not only the FOSDIC scanner, but also

continued

MAN AND COMPUTER *continued*

the communications facilities, displays, pen plotters, and multiple tape units serving the system. Although the processor is a relatively slow computer, it is useful because of its varied operations, such as table lookup and memory intertransfer. These make it adaptable for code conversion, verification, editing, reorganizing, screening, control of input-output devices, and preparation of output messages.

ACCESS is a single–address, serial binary system containing in its addressable memory over 25,000 words, each of 13 binary bits and 1 parity bit used for a quick check of the accuracy of transfer and recall. A double-length word can be used if longer ones are desired.

Four different types of memory are used in ACCESS. Transistorized registers, each containing one or two words, give fast access to a small amount of data during arithmetic and other operations; a core memory gives fast access to about 1,000 words of information; and a magnetic drum memory stores up to 24,000 words. Finally, four magnetic tape units store large volumes of information to which extremely fast access is not required. Five octal characters, composed of 15 binary bits, are used to address words in the drum, core, and register memories.

Operation of ACCESS

The FOSDIC scanner is an integral part of the ACCESS system. Although originally designed to detect the presence of marks on census forms at specified positions, it is used by ACCESS to read pictorial material as binary information and to selectively read specified areas on the film image. The Bureau in its predelivery experiments extended ACCESS's reading ability to microfilms of map tracings on coordinate paper. It matched map symbols against those of a blank master map, previously entered in the memory, to assemble frames of county maps into the State map.

Tracing the lines on a map is accomplished by a line-following program which searches for a point on a line and, when it finds one, analyzes the eight surrounding points on a fine grid to find the direction of the line. One of the two peripheral points then becomes the central point, and the process is repeated to establish the line in the computer memory. At junctions the search follows the rightmost path, returning to its starting point for closed shapes. The search reverses when it comes to the end of a line in the field, so that such a line is sensed as a collapsed contour.

The line-following technique was used in the NBS experimentation to read closed contours (such as are seen on weather maps), county boundaries, and State outlines. The data are stored on magnetic tapes as X and Y coordinates of the lines and can be produced as a trace on a display oscilloscope or as a permanent record on an X–Y digital plotter. Symbols and map contours on microfilm frames were produced on a large X–Y plotter in such positions that a complete map was assembled from microfilmed portions of it.

NBS is still experimenting with aspects and variations of the line-following program. Bureau scientists are studying recognition of the middle and junctions of lines and directing search into untried line branches. They are also looking into the effects of varying such operational parameters as the level of recognition threshold, type of instructions, and characteristics of the search area.

[1] FOSDIC II reads microfilmed punched cards, NBS Tech. News Bull. 41, 72–74 (May 1957), and FOSDIC III to assist in 1960 census, NBS Tech. News Bull. 43, 106–107 (June 1959).

[2] The AMOS IV computer for a prototype automatic weather station, NBS Tech. News Bull. 45, 13–15 (January 1961).

STANDARDS AND CALIBRATION

CHANGES IN NBS RADIO BROADCASTS

In accordance with the Bureau's policy of giving monthly notices regarding changes of phase in seconds pulses, notice is hereby given that an adjustment was made in the phase of seconds pulses emitted from radio station WWVB, Fort Collins, Colo. On 1 March 1966, the clock at the station was retarded by 200 ms at 0000 UT (7 p.m. EST of February 28). The successive time pulses emitted from station WWVB are 1 second apart. The carrier frequency is 60 kHz and is broadcast without offset.

Notice is also hereby given that no adjustment was made in the phases of time signals emitted from radio stations WWV, Greenbelt, Md., and WWVH, Maui, Hawaii, on 1 March 1966. During 1966, the pulses will occur at intervals which are longer than 1 second by 300 parts in 10^{10}, owing to the offset to be maintained in carrier frequencies, as coordinated by the Bureau International de l'Heure.

Phase adjustments, when made, insure that the emitted pulses from all stations will remain within about 100 ms of the UT2 scale. They are made necessary because of changes in the speed of rotation of the earth with which the UT2 scale is associated.

Daily UT2 information is obtained from weekly forecasts of UT2 provided by the U.S. Naval Observatory in accordance with the close cooperation maintained between the two agencies.

Wolfgang Haller uses a hypodermic syringe to inject test substances into the new-type chromatographic column. The hypodermic needles connect the eluent supply and collector to the column.

Rapid Separation Of Macromolecules And Virus Particles

A new type of chromatographic column material, consisting of nonswelling grains of a high-silica glass, has recently been studied at the NBS Institute for Materials Research. Chromatographic columns filled with the new material, developed by staff member W. Haller,[1] can separate molecular-size susbtances larger than those separated by organic-gel columns presently used in steric exclusion chromatography.

Test results indicate that such columns should be useful for the separation of macromolecular substances, virus particles, and cell components, particularly in large-scale or sterile operations where speed, reproducibility, and reprocessing of contaminated bed material is of benefit. Easily controlled and measured pore size and narrow pore-size distribution further suggest the use of the column material for size determination in analytical and diagnostic procedures.

The new column material offers other advantages not obtainable with previous techniques. For example, it will not deform under its own weight, nor under hydraulic or osmotic forces. This feature provides completely reproducible column packings and very low flow resistance, independent of flow rate or pore size. The glass grains do not contract on drying, and effective pore sizes can be checked by electron micrography or other techniques. The glass is not affected by oxidizing or acidic solutions, nor by heat. The column can therefore be sterilized as well as reprocessed after contamination with organic substances.

These advantages result from two major features of the column material. First, since it is glass, it is inert to heat and most chemicals. Secondly, unlike a gel, its pore size can be controlled over a wide range without affecting the pore volume or the rigidity of the glass. A technique to closely control pore size was developed by Dr. Haller while studying the rearrangement kinetics of the microheterogeneous regions in alkaliborosilicate glasses.[2]

In the present study, artificial mixtures of plant viruses and bovine serum albumin were used to investigate the separation capacity of the columns. Columns of 41 cm^3 were filled with 24 g of porous glass (dry weight) which ranged in pore diameter from 170 to 1700Å. The pore volume of such glass is 50 ± 3 percent and its apparent density is 1 g per cm^3. Hence, dead space is 17 cm^3 and the total free volume is 29 cm^3. The column ends were enclosed with screw-type closures containing perforated plates (glass or stainless steel) and rubber membranes.

To achieve flow of eluent through the column, the rubber membranes were punctured with hypodermic needles connected to the eluent supply and the collector. Test substances were similarly injected into the head end of the column through the membrane with a hypodermic needle and a syringe. Less than a 0.5 m head was needed to establish a flow rate of 1 cm^3 per min in a column 1 cm^2 in cross section because of the low flow resistance of the column. Flow rates as high as 5 cm^3 per min and above can be obtained with pumps.

In the tests, substances larger than the pores passed through the column and peaked at 17 cm^3, which agrees with the dead space of the column. Substances very much smaller than the pores peaked at 29 cm^3, which agrees with the total free volume of the column. The peak positions were reproducible from column packing to column packing and complete fractionations were obtained within 10 minutes.

During the tests, contaminated columns were washed with hot nitric acid and flushed with water without affecting the separation characteristics of the column material. The columns may thus be re-used time and again, a feat not possible with other devices.

[1] For further information, see Chromatography on glass of controlled pore size, by W. Haller, Nature 206, 693–696 (May 15, 1965).

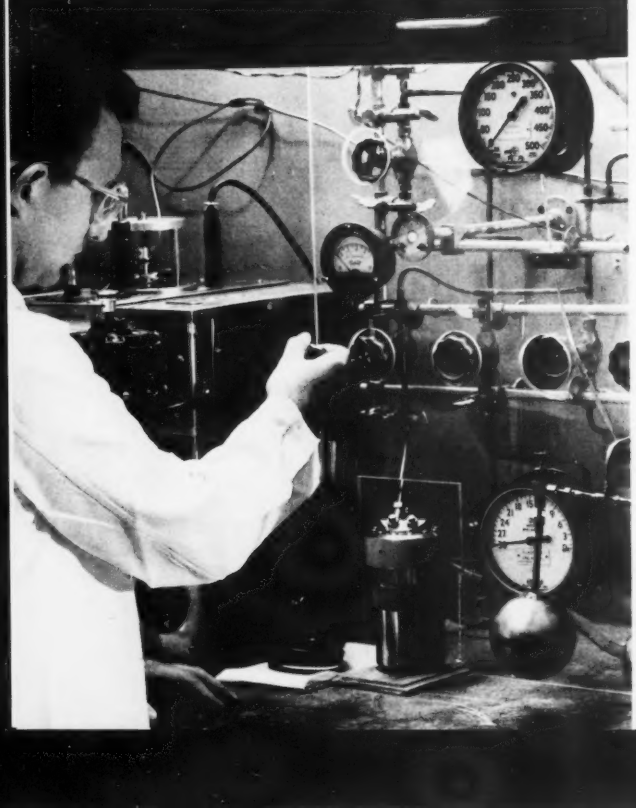
[2] Rearrangement kinetics of the liquid-liquid immiscible microphases in alkali borosilicate melts, by W. Haller, J. Chem. Phys. 2, No. 2 (January 1965).

Determination Aids Missile Propellant Studies

Heat of Formation of Aluminum Fl

Use of fluorine bomb calorimetry has resulted in the first successful determination of the heat of formation of aluminum fluoride by direct combination of the elements. This investigation was carried out by Eugene S. Domalski and George T. Armstrong of the NBS Institute for Basic Standards.[1] The heat of formation of aluminum fluoride is important for accurately predicting the effectiveness of solid propellants containing aluminum. The propellants combine with oxidizers containing fluorine. Aluminum is potentially useful as a solid-propellant constituent because of its high heat of combustion in fluorine per unit weight.

Eugene S. Domalski adjusts a fluorine inlet valve to the combustion bomb (below his hand). Fluorine inlet pressure is read on a gage (upper right). When the bomb is filled, it is removed from the line and placed in the calorimeter (box, background). A sample of the product gases in the bomb is collected in a bulb (in front of the gage, lower right).



Substitution of fluorine for oxygen in bomb calorimetry has been an important step in determining the heats of formation of metal fluorides more accurately. In this method, metals react directly with fluorine to produce the metal fluoride; thus, the determination is independent of errors introduced by auxiliary heat-of-formation data. However, difficulty in determining the best arrangement (optimum combustion conditions) for the reacting substance, difficulty in manipulating fluorine (an extremely toxic and reactive gas), and lack of fluorine of sufficient purity have delayed widespread utilization of fluorine bomb calorimetry.

In the NBS determination of the heat of formation of aluminum fluoride, the combustion bomb used was made of nickel, which is nearly inert to fluorine, and the sample rested on a monel (nickel-copper alloy) disk at the bottom of the bomb. A manifold constructed chiefly of monel was used to fill the bomb with fluorine (99 percent).

The sample to be burned in fluorine was prepared by pelleting a mixture of aluminum and Teflon powders.[2] In preparation for a typical measurement, the bomb containing the sample was first evacuated and then filled with fluorine to a pressure of 21 atmospheres. After the excess fluorine was removed from the manifold (by flushing with helium) and absorbed in a soda-lime trap, the bomb was detached from the line and placed in the calorimeter. The sample pellet was ignited electrically by means of a tungsten fuse wire.

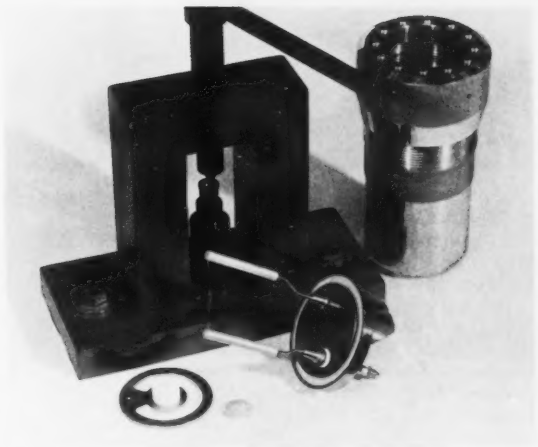
Combustion of the sample pellet was 99 to 100 percent complete. The aluminum fluoride produced in the combustion bomb was deposited mostly as a fine white powder on all surfaces inside the bomb with the bulk of the product concentrated in the immediate combustion area. Gaseous carbon tetrafluoride was the only major product of the combustion of Teflon in fluorine. Other fluorocarbons were present to less than 0.05 percent.

As a result of the NBS investigation, the heat of formation of aluminum fluoride, determined by direct combination of the elements for the first time, was found to be -1507.8 kJ/mole or -360.37 kcal/mole with an overall uncertainty of 6.6 kJ/mole or 1.6 kcal/mole. This value is significantly more negative than that previously determined indirectly by measuring the heat of reaction of aluminum with lead fluoride,[3,4] and suggests that direct combination of the elements should be used where pos-

umFluoride

sible to determine heats of formation of fluorine compounds.

[1] For further details, see Heat of formation of aluminum fluoride by direct combination of the elements, by Eugene S. Domalski and George T. Armstrong, J. Res. NBS 69A (Phys. & Chem.), No. 2, 137 (March–April 1965).
[2] Combustion and reaction calorimetry, NBS Tech. News Bull. 46, 61 (April 1962).
[3] P. Gross, C. Hayman, and D. L. Levi, Trans. Faraday Soc. 50, 477 (1954).
[4] V. P. Kolesov, A. M. Martynov, and S. M.. Skuratov, Zn. Norgan, Khim 6, 2623 (1961).



Fluorine bomb (right) and pellet press (left). The calorimeter bomb head is shown in the foreground.

NEW MASS SPECTROMETRIC DATA CENTER

Scientists and engineers may now obtain up-to-date information on the energetics of gaseous ionic processes from the National Bureau of Standards. A new facility, part of the National Standard Reference Data System,[1] furnishes data in convenient tabular form on ionization and appearance potentials and heats of formation of ionic species.

Named the Mass Spectrometric Data Center, the facility is staffed by Fredric N. Harllee, H. M. Rosenstock, and John T. Herron of the NBS Mass Spectrometry Section. Requests for data on specific ionic processes may be made either by letter or by telephone.

These data are used by physical chemists, molecular physicists, and engineers working in aerospace research, and in other fields such as petroleum chemistry, radiation chemistry, catalysis, and the synthesis of new materials. The cutoff date of the last comparable data compilation was 1955.[2] Since that time, a wealth of new material has appeared in the literature. Virtually all of this material has now been abstracted at NBS and stored on punched cards which are updated as new data are found. Current information on a particular ionic system can be made quickly available to individual investigators, thus eliminating the need for personal literature searches.

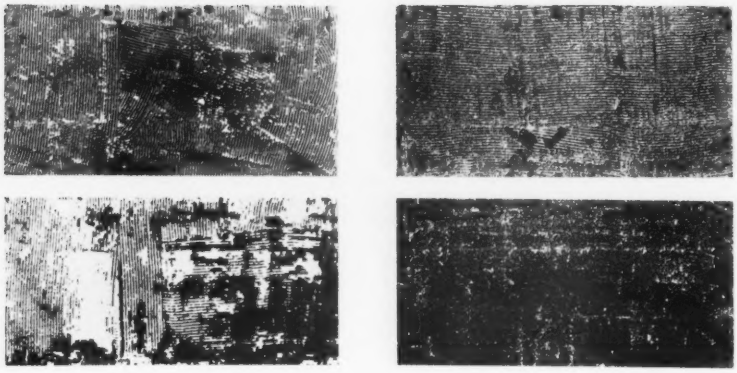
Ultimate aim of the new Center is the development of a critically evaluated and, hence, reliable body of information on fundamental molecular properties and processes that are measured principally by mass spectrometric techniques. As a first step in this direction, the Center has systematically collected and abstracted the existing literature on the energetics of ionic processes and has incorporated the resulting data into an effective retrieval system.

The NBS Office of Standard Reference Data has let a contract to Rice University, Houston, Tex., for the initial critical evaluation work, using the data now in the files of the Center. The work will be carried out by J. L. Franklin, Welch Professor of Chemistry at the University and internationally known as an authority in the field of mass spectrometry. Professor Franklin will be assisted by F. H. Field of the Esso Research and Engineering Corp., which is sponsoring Dr. Field's share of the work.

When the evaluation is completed, probably within the next 12 to 15 months, it will be added to the National Standard Reference Data Series of Publications.[1] The Center will then keep the evaluation up to date as new data on ionic processes become available.

The Center is at present engaged in a project to collect available data on the mechanisms and cross sections of ion-molecule reactions. In addition, plans are being made to collect available data on isotopic abundance determinations and electron affinities. In the meantime, special tables and bibliographies in current demand will be issued as NBS publications.

[1] National standard reference data system—plan of operation, NSRDS-NBS 1, December 1964, by Edward L. Brady and Merrill B. Wallenstein, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402 (15c); also, National standard reference data system, NBS Tech. News Bull. 47, 138 (1963).
[2] Electron impact phenomena and the properties of gaseous ions, by F. H. Field and J. L. Franklin, Academic Press, Inc., New York (1957).

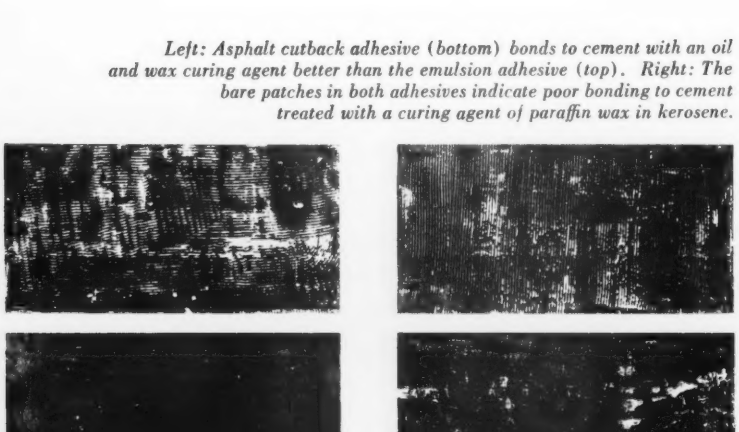


Left: Sanding a waxed surface (top) improves the bond of asphalt emulsion adhesives; an unsanded surface is at the bottom. Right: Both cutback and emulsion adhesives bond well to cements with butadiene-styrene copolymer curing agents.

Bonding of Tile and Paint

TO TREATED CONCRETE

Left: Asphalt cutback adhesive (bottom) bonds to cement with an oil and wax curing agent better than the emulsion adhesive (top). Right: The bare patches in both adhesives indicate poor bonding to cement treated with a curing agent of paraffin wax in kerosene.



The NBS Institute for Applied Technology has recently completed an investigation of the adhesion of resilient tile flooring and paints to concrete surfaces treated with curing or parting agents. In this study, sponsored by the Army, Navy, and Air Force, W. C. Wolfe found that asphalt cutback adhesive (a solvent-type asphalt adhesive) may be used successfully over most treated concrete surfaces. However, asphalt emulsion adhesive requires an asphalt primer to insure adhesion, particularly when used over oily or waxy material. Paints formulated with butadiene-styrene copolymer or chlorinated rubber in organic solvents appear to have the best adhesion to treated concrete. For good adherence, most other paints must be compatible with the treating agent.

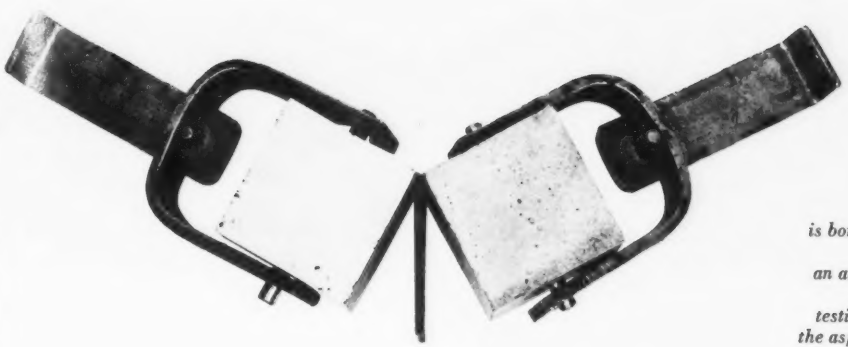
Curing and parting agents are widely used on concrete structures. Curing agents are organic materials that are applied to seal in the moisture needed to cure the concrete. They are generally solutions of synthetic resins in organic solvents; waxes, oils, silicones, or a combination of these may also be present. Parting agents are used to separate slabs in lift- or tilt-slab construction, and usually contain oils and waxes.

The failure of some coating materials to bond with concretes treated with these agents has generated considerable interest in the effect of the treating agents on adhesion. The present study was initiated to determine which coating materials may be used on treated concrete surfaces without adverse effects, and to find methods of correcting unsatisfactory surface conditions.

In addition to curing and parting agents, deposits of wax, oil, grease, or dirt may be found on concrete floors. Also, oil from molding forms may remain on concrete walls and cause trouble with paints.

Adhesion of Resilient Tile

In this investigation, asphalt and vinyl asbestos tiles that are widely



These special yokes are attached to a wooden block (left) and a cement block (right) with a piece of tile in between. After the tile is bonded to the wood with epoxy cement and to the cement with either an asphalt cutback or emulsion adhesive, the yokes are fitted to a tensile testing machine. The bond strengths of the asphalt adhesives are then determined.

used for commercial and Government resilient flooring were affixed to cement specimens with asphaltic adhesives. The adhesion of these tiles was then tested both qualitatively and quantitatively.

In the qualitative bond tests, six cement mortar panels, 5/8 by 27 by 28 in., were cured with various compositions and one panel was moist cured. Nine 9- by 9-in. tiles were cemented on each panel, with either cutback or emulsion adhesive.

The tiles were removed by hand about 3 months later, and the difficulty of removal was noted. Generally, the tiles had to be pried loose with a putty knife. No oozing between tiles was noted, even on oily and waxy panels. Light sanding of a panel treated with paste wax improved the adhesion of asphalt emulsion adhesive.

The moist-cured panel's white appearance suggested efflorescence. Tiles bonded to this panel with the cutback and emulsion adhesives were pried up with moderate difficulty, indicating fairly good adhesion. About half of the concrete surface under each tile, however, was bare. This indicated fair or poor adhesion. The bare surfaces appeared "dusty" and the adhesive remaining on the tiles was white; the adhesive apparently removed some laitance* from the concrete.

In the quantitative straight-pull bond tests, 2-inch-square asphalt tiles were bonded under pressure to the trowel-finished side of 2-inch cement mortar cubes cured under water or with various curing agents. Wooden cubes, cemented to the top of the tiles, and the mortar cubes were secured to special yokes clamped in a tensile testing instrument. Straight-pull tests were performed on sufficient cube assemblies to obtain significant results on the adhesive bond strength. Bond strength of the two adhesives differed significantly, the emulsion type having the stronger bond.

The bond strength of the emulsion adhesives was decreased by organic curing agents based on butadiene-styrene copolymer, chlorinated rubber, petroleum hydrocarbon resin, polyvinyl toluene-butadiene copolymer, paraffin wax, or paraffin wax with kerosene. None of these agents affected the bonding of cutback adhesives. Prior treatment of the concrete with sodium silicate solution did not noticeably improve the bond strength.

Adhesion of Paints

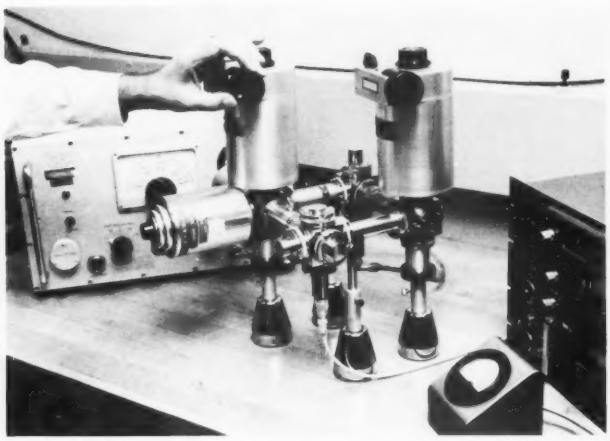
In the paint-adhesion tests, treated cement mortar panels were coated with a variety of paints. Adhesion was judged qualitatively after 1, 7, and 30 days by scraping with a sharp knife and by a pressure-sensitive tape test. In this test, a cross about ½ by 1 in. was cut in the surface of the paint, and tape was applied over the cross. The tape was detached with a sharp pull as nearly as possible in the plane of the surface and the amount of paint removed was observed.

Cement-water paints adhered poorly to concrete treated with curing agents, because they need a rather porous hydrophilic surface. Other types of concrete paints had excellent adherence to panels cured with agents based on either a butadiene-styrene copolymer or chlorinated rubber, but varied in compatibility with other curing agents. In general, emulsion or latex paints did not adhere well to panels treated with oily or waxy materials. Organic solvent-based paints were generally satisfactory when applied over oils and waxes. Paints formulated with butadiene-styrene copolymer or chlorinated rubber in organic solvents, such as xylene or mineral spirits, appeared to have the best adhesion.

*Laitance consists of small particles of material that form a whitish scum on the surface of fresh cement.

210-113 O—66——2

The twin-T bridge shown has been assembled from modules developed at the NBS Radio Standards Laboratory. Here the twin-T is being used to calibrate an incremental conductance standard for use with admittance ratio bridges.



MODULAR TECHNIQUE GIVES ACCURATHF

A modular method of building high-frequency electronic equipment, developed by the Radio Standards Laboratory, has led to instruments which are both more accurate and more versatile than conventional units. The new technique has been adapted to a number of impedance- and admittance-measuring devices by Leslie E. Huntley and Robert F. Metzker of the Radio Standards Engineering Division, and can be applied to devices for measuring other electrical quantities.

Like a child's Tinker Toy, the modules can be readily joined to form a variety of measuring instruments.[1] Thus it is possible to assemble a special calibration instrument without going to the expense and incurring the delay of custom design and construction. Each component or module can be defined and evaluated individually, making possible more economical calibration service facilities and improvement in the accuracy of the overall instrument.

An additional advantage of the modular construction technique is that in the event of component failure, the malfunctioning module can be replaced with a preadjusted, precalibrated one and the instrument can be returned to service with a minimum of costly downtime. The same is true of instruments needing recalibration of a module for continued accuracy. Obsolescence of measuring instruments is prevented because any advancement in the accuracy of a standard improves the accuracy of all instruments using the module since the module is the standard. Further, the capability of repeating measurements, with exchange of immittance* standards and unknowns, facilitates statistical evaluation of the calibration process.

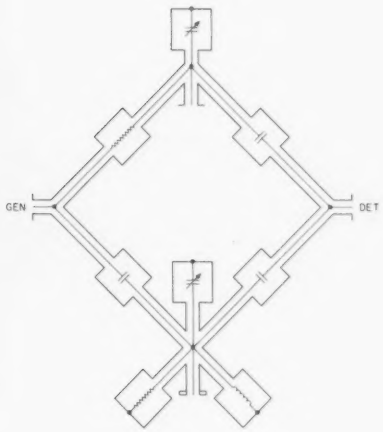
Modular Technique

Key to the modular construction is a sexless, precision coaxial connector which has a definable reference plane and can be mated, without adapters, with any other connector of the same size. This makes it possible to assemble standard components in a variety of combinations unlimited by connector requirements. Connectors of three designs are used for this purpose at NBS: one is a 19-mm connector adapted from a design introduced in England;[2] the other two are the 7- and 14-mm connectors proposed as IEEE standards. The following modules have now been built at the Bureau: one- and two-port resistors, capacitors, and inductors; one-port incremental capacitors, resistors, and short circuits; and six-port junctions.

Instruments Built

One of the instruments recently built is an admittance

W. F. Clore takes a final cut on a 7-mm cube connector (six-port coaxial junction), used to connect together several modules designed at the NBS Radio Standards Laboratory.



Highly precise machining techniques were used to fabricate the modules assembled in this twin-T bridge on the right. A diagram of the bridge is given on the left.

ATHF INSTRUMENTS

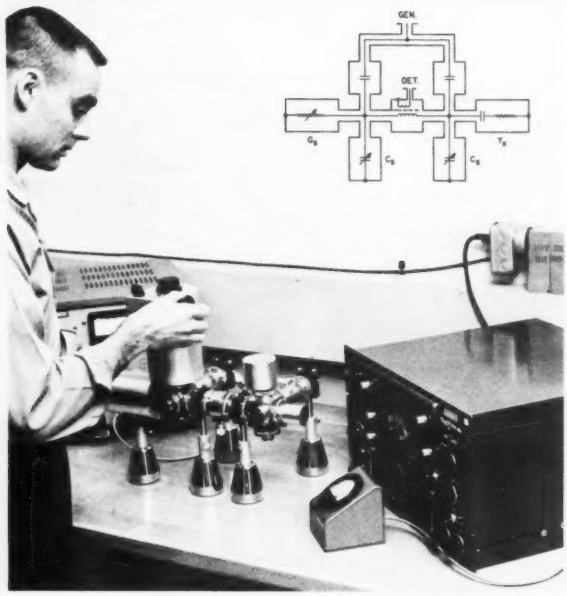
bridge that provides for selection of the admittance range by substitution of the appropriate standard as a ratio element, in contrast to the circuit-switching methods of conventional instruments. Elimination of switching results in circuit simplification. Also, network effects resulting from the presence of unused ratio arms are eliminated and frequency characteristics are improved. The accuracy of a modular instrument can be established and maintained easier than that of its conventional predecessor since it is composed of standards easily removed for calibration. Finally, the unknown admittance is determined with maximum accuracy by balancing it against a high quality fixed standard of about the same value, in parallel with small incremental standards.

Another instrument constructed by this technique is a self-calibrating twin-T instrument having 19-mm connectors.[3] This instrument is used to measure conductance in terms of capacitance throughout the HF range (to 30 MHz). A similar instrument with 7-mm connectors has been operated over the VHF range. Still another, employing a series resonance technique for determining absolute conductance, was used at 100 kHz to verify Q measurements made with the twin-T instrument.

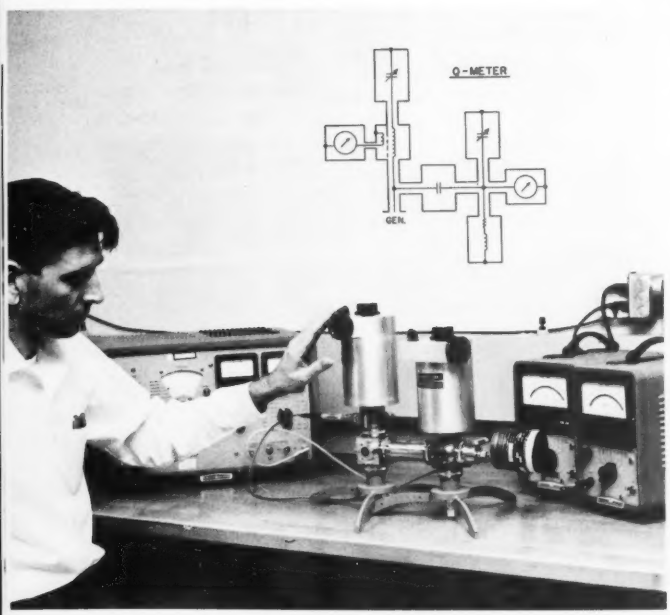
The Bureau's promising experiences with calibrated modules may encourage further use of this approach in

continued

View of 7-, 14-, and 19-mm connector pairs used in NBS instrumentation. The size refers to the inside diameter of the outer conductor.



Robert F. Metzker uses the NBS modular admittance ratio bridge, assembled according to the diagram shown above, to measure a fixed capacitor.



NBS engineer Leslie E. Huntley makes measurement needed to determine the conductance and resonating capacitance in a series-resonant circuit. The equipment has been assembled in the configuration shown above.

MODULAR TECHNIQUES *continued*

designing and producing instruments. The modular technique could enable instrument manufacturers to meet present demands for calibrated immittance-measuring instruments.

Design Data

In an admittance ratio bridge, the required admittance range can be obtained by the use either of multiple ratios and a small number of decades in the standard arm, or of a single fixed ratio and a large number of decades in the standard arm. In conventional, hand-wired ratio bridges, multiple ratios are usually obtained by switching circuits; this practice increases the residual shunt admittance and series impedance of the ratio elements. In modular construction, the ratio elements are individual standards that can be freely substituted, so the need for internal switching circuits is eliminated and network effects that degrade frequency characteristics are reduced.

If a large number of decades are used to obtain a wide admittance range, the residual admittance in the standard arm of the bridge is thereby increased, making it necessary to increase the "zero" admittance in the unknown arm to obtain initial balance. Because the standard admittances are physically large, the residual impedance between the standard elements and the bridge corners is large. An increase of the residual admittances in the standard and unknown arms tends to decrease the bridge sensitivity and cause stability problems. The impedance in series with the standards makes necessary large corrections to the bridge readings at the higher frequencies.

In modular construction, high-quality fixed standards of the appropriate value of the unknown admittance are connected in parallel with small incremental standards at the bridge corner. The unknown admittance is then measured in terms of a small difference from the externally connected fixed standards. Since the initial balance is obtained with the small incremental standards, the "zero" admittance is minimized. The high frequency corrections to the bridge readings are minimized because the compact design of the immittance modules decreases the residual impedance in the standard arm.

*The word "immittance" is a composite of impedance and admittance. It stands for impedance and/or admittance.

[1] This program should not be confused with Project TINKERTOY, which was conducted by the Bureau in the early 1950's to demonstrate the feasibility of machine-producing vacuum-tube modules. See Project TINKERTOY, modular design of electronics and mechanized production of electronics, NBS Tech. News Bull. 37, 161–170 (November 1953).

[2] A coaxial connector system for precision R.F. measuring instruments and standards, by D. Woods, Proc. IEEE, 108B, Paper No. 3499E, 205–215 (March 1961).

[3] A self-calibrating instrument for measuring conductance at radio frequencies, by Leslie E. Huntley, J. Res. NBS 69C (Eng. and Instr.), No. 2 (April–June 1965).

WAVELENGTH DEPENDENCE IN THERMOPILES OBSERVED

Photoelectric Effect Causes Errors in UV Radiation Measurements

Accurate measurement of detected radiation in the far ultraviolet is of great importance in space physics and astrophysics. Knowledge of the radiations found in outer space is needed to design and construct long-range space vehicles and to provide adequate protection for persons traveling in these vehicles. Thermopiles—a number of thermocouples connected in series—are often used to detect radiation in the far UV spectral region. Because difficulties arise in making calibrations in the UV, the thermopiles are generally calibrated with visible and near-infrared radiation, on the assumption that the sensitivity of the thermopile will not change as the wavelength of the radiation changes.

As one phase of a program to establish radiation standards in the UV region, studies to test the "wavelength independence" assumption have been made by R. G. Johnston and R. P. Madden of the NBS Institute for Basic Standards,[1] under the sponsorship of the National Aeronautics and Space Administration. In one of these studies, a significant wavelength dependence was observed and traced to the photoelectric effect. This finding indicates that corrections should be made for thermopiles calibrated in the visible but used to detect radiation in the far ultraviolet.

The study was carried out by means of a vacuum ultraviolet monochromator with a 1-meter radius and a 600-line/mm concave grating. The radiation source was a windowless d–c capillary discharge tube. The thermopile consisted of a series connection of four elements composed of bismuth-tellurium and bismuth-antimony alloys.

In the first test with this equipment, the scattering of incident radiation as a function of wavelength was investigated. It was found that a small amount (2–4 percent) of the incident radiation was scattered (reflected away), but this occurred in all cases, regardless of the wavelength of light used. A second test indicated that no wavelength dependence was created when alternating current was used in place of direct current to amplify the radiation source.

A third test, however, did reveal a significant wavelength dependence. It was found that as ultraviolet radiation of wavelengths below 1600 Å strike the black absorbing material of the thermopile, the energy of collision causes electrons in the black material to be ejected.

Apparatus used to study the sensitivity of thermopiles in detecting far UV radiation. Measurements take place in the steel chamber (center right) which is connected to a vacuum monochromator (lower left). The monochromator generates ultraviolet light of particular wavelength.

This photoelectric effect occurs only with short-wavelength ultraviolet light; the longer wavelengths of visible light cannot produce it. The ejected electrons carry away a significant amount of the incident ultraviolet energy (as much as 5 percent), thus resulting in a substantial variation between ultraviolet and visible light measurement.

These tests were conducted with a particular type of thermopile and blacking material; however, the ones chosen are typical of those commonly used in the ultraviolet region. The particular significance of the work is that the wavelength dependence and its cause have been definitely established and the necessity for a correction has been noted. It appears that if the necessary corrections are determined carefully for an optimum thermopile, a radiation detection standard could be established for the far ultraviolet. Such a standard would be reliable to several percent—a very desirable goal at this time. Work is now continuing in this area.

[1] For further technical details, see On the use of thermopiles for absolute radiometry in the far ultraviolet, R. G. Johnston and R. P. Madden, J. Appl. Optics 4, 1574 (1965).

Pressure Transducer Response

Affected by Thermal Gradients

Recent research at the NBS Institute for Applied Technology disclosed that the performance specifications of many flush-diaphragm pressure transducers do not describe their actual performance when they are subjected to thermal gradients. The degradation of performance is temporary; the response typically returns within its specified limits when thermal equilibrium is regained. This aberration was studied by physicist Leon Horn of the Institute's instrumentation laboratories. It is detected by a procedure he developed for testing transducer performance under controlled conditions of thermal shock.[1]

For many years the Bureau has investigated transducer problem areas, one of which is the validity of performance specifications under steady-state conditions. Pressure transducers have been assumed to operate correctly if zero-shift and sensitivity remained within specified limits during certain tests conducted under static temperature conditions. Dynamic temperature effects were almost ignored. Increased use of transducers in the presence of thermal gradients, as in rocket-motor and space-environment tests, now makes it necessary to verify instrument performance under dynamic temperature conditions.

Changes in ambient temperature can affect pressure gage performance by shifting zero values, altering sensitivity, or changing damping. Manufacturers have attempted to compensate for static changes in designing instruments. Compensation for dynamic thermal changes is not always possible, however, as each component responds to a thermal wave at a time determined by the path length and the conductivity of the materials between it and the heat source. Therefore, some shift in the zero indication of any pressure transducer must be expected in the presence of thermal gradients.

Testing Response to Gradients

The test procedure developed by Mr. Horn for determining transducer response uses a device which applies an accurately known temperature gradient to the transducer while its output is being monitored. The device consists of a heating element surrounding a well holding fused, low-melting-point metal. The temperature, continuously controlled by an autotransformer between 225–900 °F (107–482 °C), is accurately known by means of a thermocouple. The transducer being tested is mounted on a platform that is swung down so the transducer just touches the molten metal, at which time the temperature gradient across the transducer is measured.

Transducer temperatures during dynamic testing, obtained from thermocouples in the molten metal and at the rear of the transducer, are permanently recorded on a strip chart. Transducer output data are recorded by photographing the oscilloscope presentation of transducer output for sweep durations of the order of minutes.

Findings

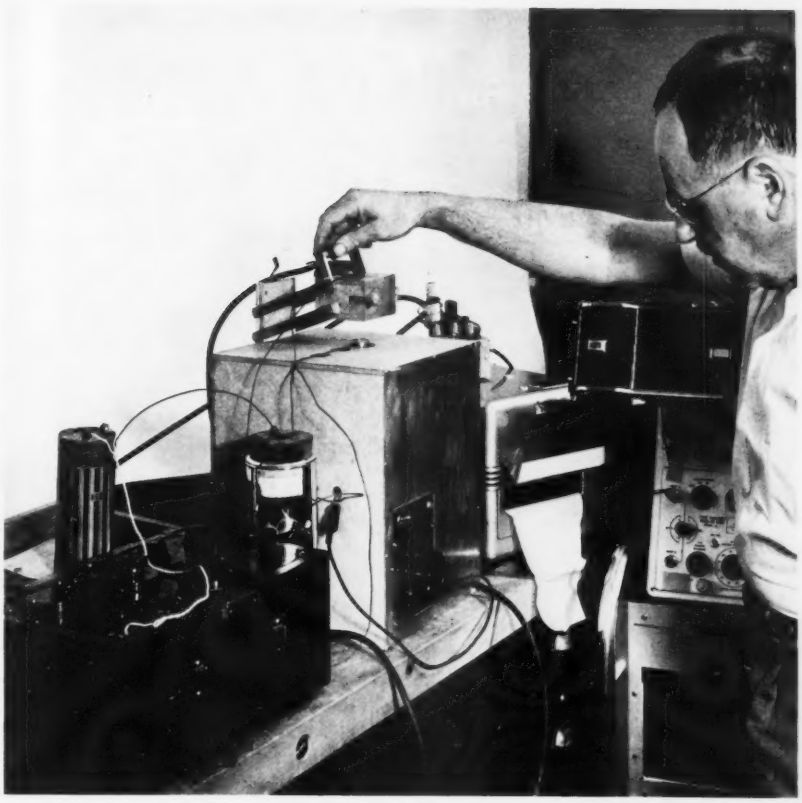
Thermal response tests on flush-diaphragm type pressure transducers showed that the response in the presence of gradients could not have been anticipated from device specifications. The zero-shift of some transducers was within specified variability but for others it far exceeded the specified limits. For some instruments a zero-shift of 10 percent of the full-scale pressure indication is allowable for a temperature change from room temperature to 600 °F (an increase of about 320 °C). Mr. Horn found shifts of 40 percent for some transducers subjected to this thermal change. Instruments having such characteristics may be suitable under stable conditions, but completely unacceptable in the presence of large thermal gradients.

The typical zero-shift consists of an initial shift due to the increased temperature of the transducer face, followed by a rapid recovery and overshoot of greater amplitude and duration. The overshoot builds up to a maximum as the temperature gradients sweep across the structure and then decays asymptotically. Time required to reach a maximum—a minute or less—is determined by instrument materials and design. Ultimately, when all gradients have passed, the transducer components are once again at the same temperature and the instrument's indication returns to the zero for that temperature.

Attempts to categorize pressure transducer behavior according to design type (strain gages on diaphragm, strain gage stressed by linkage, piezoelectric, bellows, and differential transformer, for instance) have been unsuccessful. Apparently, details of design and selection of materials are more important than is the type of construction in determining thermal characteristics.

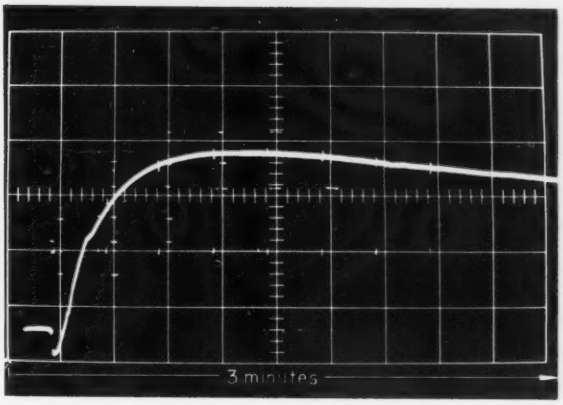
Categorization by manufacturer was also useless in predicting behavior. Comparison of units of the same manufacture and design series showed one to have a desirable thermal characteristic, while another, subjected to a sudden gradient, shifted its indication by more than 100 percent of full scale and became inoperative in less than 20 sec. After removal of the gradient, the transducer recovered and performed satisfactorily. A third gage of this series recovered sufficiently for the zero indication to return within the specified limits within 40 sec.

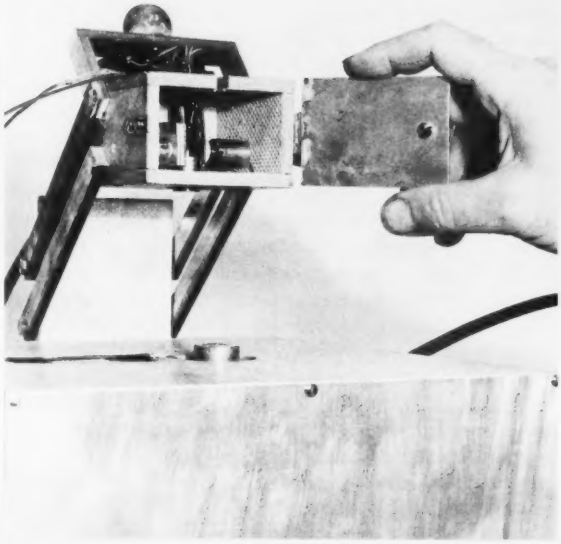
[1] The response of flush diaphragm pressure transducers to thermal gradients, by Leon Horn, Preprint No. 13.3–4–65, 20th Annual ISA Conference, Oct. 4–7, 1965, Los Angeles, Calif. (Available from Instrument Society of America, 530 William Penn Place, Pittsburgh, Pa. 15219, for 75 cents.)



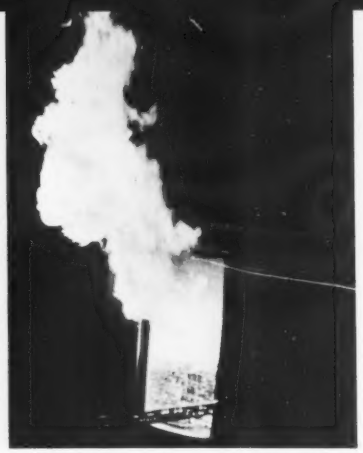
NBS scientist Leon Horn tests a pressure transducer's response to thermal shock by bringing the transducer (not visible) down to touch the molten metal below it. Thermocouples at the metal and the transducer are referenced to junctions in an ice bath beside the potentiometer at left.

Below, the slow-sweep (3-minute) oscillogram shows the initial shift in zero-pressure indication of a pressure transducer subjected to a thermal shock. It immediately swings back and far overshoots the zero position, reaching a maximum in less than a minute. (Large vertical divisions = 10 percent transducer full scale.)





Left, the transducer protruding through the bottom of this box will be swung down so that its bottom is in thermal contact with the molten metal in the well below, which is at a known temperature. Temperatures of the transducer and the molten metal are measured by thermocouples (leads from left) and variations of the transducer output are analyzed to determine its response to thermal gradients.



Flaming is stable in this small-scale model used in a recent study of enclosed fires. By varying the size of the window opening, ventilation parameters were defined which hopefully may be used to relate data on model fires to fires in full size structures.

SCALING FACTORS FOR FIRES
In Test Models

Large-scale fire research experiments are expensive and difficult to analyze because of the interactions of mechanical, chemical, thermal, and diffusional processes. Although these interactions also complicate analysis of fires in small-scale models, the use of such models under controlled conditions may provide useful information at much less cost. To facilitate their application, the NBS Institute for Applied Technology initiated a study to develop basic information on the use of small-scale models in fire research.

In this study,[1] D. Gross and A. F. Robertson investigated three different sizes of model enclosures. They found scaling relationships between the sizes which may be useful in relating the models to full-scale structures. The scaling relationships were obtained by "normalizing" the burning rate and ventilation parameter.

Experimental Models

The models used in this study were three geometrically similar box-type enclosures in each of which the width, height, and length dimensions were in the ratio 1 : 1 : 2. Model widths were 16.5, 46, and 145 cm, for the three enclosures, designated I, II, and III, respectively. Each model had a single ventilation opening or "window" centrally located on the front end. For vertical windows, the opening was full height and adjustable in width from closed to fully open. The opening of the horizontal windows was full width and adjustable in height from closed to fully open.

Reproducible crib-type combustible loads were used in the models. The cribs consisted of sticks of unfinished cellulose-base fiberboard having high flammability and uniform burning characteristics. They were supported above the floor between the center and rear of the models and were ignited by kerosene-soaked fiberboard wicks.

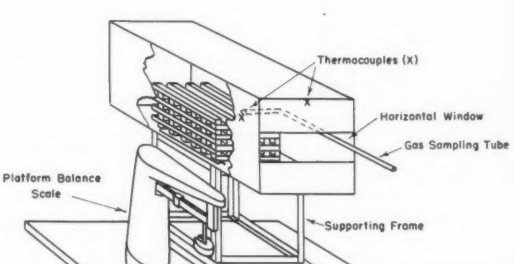
Although mass burning rates, temperatures, and gas compositions were measured for the duration of each test, only the data recorded for fully developed fires were considered. These data correspond to the period of burning during which crib weight decreased from 80 to 30 percent of its original weight.

Findings

In all three models, as ventilation was increased gradually, the fully developed fires had two reactions separated by a transition zone. The first reaction was essentially a smoldering one with little visible flame, as ventilation was not sufficient to permit stable flaming within the enclosure. The burning rate increased with increased ventilation until the transition zone was reached. Here the burning rate no longer increased with ventilation and in some instances the burning rate actually decreased. After the transition zone the burning rate again increased with increased ventilation and flaming became stable. Finally a point was reached where further ventilation increases had no effect. Burning was then no longer ventilation limited, but corresponded to crib burning in open air (free burning).

Cribs of identical weight, construction, and exposed surface area were used in tests of model I and in most tests

In the experimental apparatus used for fire research at the NBS Institute for Applied Technology, crib-type loads in small-scale model enclosures were supported on a platform balance scale. Mass burning rates, temperatures, and some gaseous combustion products were measured in fully developed fires as the crib weight decreased from 80 to 30 percent of the initial weight.



of model II. In other tests of model II and in tests of model III, some weight and surface variations were introduced in the cribs. The results indicated that the burning rate and the ventilation parameter (the window area times the square root of the window height) of a model could be related to those of another model by dividing the parameters by the square of the linear scale ratio. With model II as the reference, "normalized" parameters were obtained for models I and III.

The normalized burning rates were plotted against the normalized ventilation parameters for each model. Visually estimated "best fit" lines were drawn through the points plotted above the transition zone, and lines parallel to these were drawn through the points below the transition zone. Horizontal lines were drawn through the points indicating free burning.

The best fit lines for the three models show an apparent overall correlation of the data despite the size, weight, and surface area differences of the models. The lines for models II and III have a slope of one, indicating a direct proportionality, whereas the data for model I indicate a higher power relationship.

As these results apply only to the models and cribs studied, caution should be exercised in attempts to apply this data to other situations.

[1] For further information, see Experimental fires in enclosures, by D. Gross and A. F. Robertson, Proceedings of the 10th International Symposium on Combustion, p. 931–942 (1965).

FIRST IMR SYMPOSIUM ON MATERIALS RESEARCH

The NBS Institute for Materials Research (IMR) will sponsor its first Symposium on Materials Research with international participation at the Bureau's new facilities in Gaithersburg, Md., October 3–7, 1966. The subject of this meeting will be "Trace Characterization—Chemical and Physical." General chairman for the event is Dr. W. Wayne Meinke, assistant general chairman is Dr. Roger G. Bates, and Bourdon F. Scribner is the technical program chairman.

The symposium will bring together leading authorities in many different fields which contribute to the physical and chemical characterization of materials, especially in the detection and determination of trace amounts of defects and "foreign" substances. Current trace characterization problems will be discussed and the successes and shortcomings of materials characterization will be examined. In addition, experimental methods and techniques will be compared and evaluated.

Thus, the symposium should improve the effectiveness of materials research by summarizing the present capabilities and future potential of the more useful tools for trace characterization, and by promoting the exchange of knowledge among the varied disciplines upon which complete and meaningful characterization depends.

Morning and afternoon sessions of the program will be held in the new NBS auditorium with the morning sessions devoted to two 1-hour invited lectures and related discussion. Shorter contributed papers will be scheduled in the afternoon, followed by discussion periods. No simultaneous sessions will be held.

Those desiring to present papers should submit titles and abstracts of about 300 words to B. F. Scribner, Institute for Materials Research, National Bureau of Standards, Washington, D.C., 20234, before May 1, 1966. These papers should emphasize new methods or capability in the chemical and physical trace characterization of materials. Critical papers on specialized aspects of the principal themes, especially those on new developments and broadened potential, will be welcome. Limited program time precludes detailed description of well-known methods and techniques.

The following general areas of trace characterization will be covered:

1. Electrical (e.g., resistivity, Hall effect, dielectric relaxation).

2. Spectroscopic (e.g., emission, x-ray, including microprobe, atomic absorption).

3. X-ray diffraction (e.g., lattice parameter, electron density, topography).

4. Optical (e.g., color center studies, light scattering, spectrophotometry).

5. Electrochemical (e.g., polarography).

6. Nuclear (e.g., activation analysis, radioisotope dilution).

7. Resonance (e.g., electron spin resonance, ESR, and nuclear magnetic resonance, NMR).

8. Mass spectroscopy (e.g., spark source, isotope dilution).

9. Electron and optical microscopy (e.g., replica and transmission techniques, electron diffraction).

10. Sample preparation, preconcentration, handling, reagents, etc.

Both invited lectures and selected contributed papers will be published in a volume to be prepared from the proceedings of the symposium. Manuscripts must be in the hands of the technical program chairman by September 1, 1966. All individuals interested in receiving later announcements concerning the symposium should write Dr. Roger G. Bates, Institute for Materials Research, National Bureau of Standards, Washington, D.C., 20234.

PUBLICATIONS

of the

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PERIODICALS

Technical News Bulletin Volume 50, No. 3, March 1966. 15 cents. Annual subscription: $1.50. 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

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Section B. Mathematics and Mathematical Physics. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75. Single copy, 75 cents.

Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic, $2.75; foreign, $3.50. Single copy, 75 cents.

CURRENT ISSUES OF THE JOURNAL OF RESEARCH

J. Res. NBS **70C** (*Engr. and Instr.*), *No. 1* (*January–March 1966*), *75 cents.*

Coulometric calibration of microvolumetric apparatus. G. Marinenko and J. K. Taylor.

Effect of temperature and notch geometry on the tensile behavior of a titanium alloy. W. D. Jenkins and W. A. Willard.

Measurement standards for low and medium peak pulse voltages. A. R. Ondrejka and P. A. Hudson.

Temperature coefficient of rf permeability measurement using an impedance bridge as an equality indicating device. A. L. Rasmussen.

A test apparatus for the study of forced air-mixing devices. T. K. Faison, J. C. Davis, and P. R. Achenbach.

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